Ultrasound- associated extraction of seed oil of Korean pine

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Abstract: Experiment on ultrasound- associated extraction of seed oil of Korean pine (*Pinus koraiensis*) was conducted in Northeast Forestry University, Harbin, China. The factors affecting extraction yield, such as ultrasonic frequency, extracting temperature, extracting time and the ratio of material to liquid (ratio of Korean pine seed to absolute alcohol), were analyzed under specific condition and the optimal extracting parameters were obtained as the ultrasonic frequency 32 000 Hz, the extracting temperature 80 ℃, the extracting time 50 min, and the ratio of material to liquid 1: 30. The study demonstrates that ultrasound is a reliable and great efficiency tool for the fast extraction of Korean pine seed oil,

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Introduction

Ultrasonic is related to acoustic frequencies above the audible range to human ear, approximately above 20 000 Hz, and it can produce a high-temperature environment within a short time, together with strong shock wave and discharge, shine, etc. The action of ultrasound is affected by many factors, such as ultrasound frequency, temperature, time, and so on. The energy produced by ultrasonic can change physical structure, state, and function or accelerate the process of changes. Commonly accepted that effect of cavitations, heat, and mechanical effect are the primary theoretic basis of ultrasonic (Zeng *et al.* 2002).

Ultrasonic extraction technology has been widely applied in extracting oil, pigment, proteins, and flavonoids in plant, etc. (Han *et al.* 2004; Li *et al.* 2003; Li *et al.* 2004; Shi *et al.* 2004). Research showed that this technology has great efficiency, uses less energy, and does not destroy the effective component of materials. The seed oil of Korean pine (*Pinus koraiensis*) is one of the most valuable natural vegetable oils. Up to now, however, the extraction of seed oil of Korean pine by ultrasound-assisted extraction technology has not been reported. In this study, the influence of the different factors on extracting yield was investigated.

Material and method

Material

Korean pine seeds were collected from Xiaoxing'an Mountains, Heilongjiang Province, China. All ultrasonic extractions were carried out by KQ Signature Ultrasonic Cleaner Model 250DB with digital timer, heat and power (Kunshan Ultrasonic Instruments Ltd., China). Electronic scale and electrothermal constant temperature blast drier were provided by Tianjin Test Instruments Ltd. Low speed centrifuge (LXJ-ⅡB) was provided by Shanghai Anting Scientific Apparatus Factory. Rotary evaporator (R-205) was provided by Shanghai Shensheng Bio-Techno. Ltd. and absolute alcohol (AR) was provided by Beijing Chemical Reagent Plant.

Method

Dried seeds (20.0 g) of Korean pine were broken into pieces, put it into a beaker, added absolute alcohol, and then treated with ultrasonic wave as plan. And the solution was separated by centrifugation (30 min, 5 000 $r \cdot min^{-1}$), and ethanol was reclaimed from the solution by rotary evaporator as possible. Finally, the oil was dried at 105℃ for about 2.0 h (the weight was invariableness) by the drier, then cooled, and weighed. The yield of extraction is calculated by the following formula:

Extraction yield= Extraction weight / seed weight × 100%

Results and analysis

Effect of ultrasonic frequency on extraction yield

The effect of different ultrasonic frequency on extraction yield was detected at 20-min ultrasonic time, the ratio of material to liquid (ratio of Korean pine seed to absolute alcohol) of 1:10, and temperature of 40 ℃. Extraction yield was highest at ultrasonic frequency of 32 000 Hz, and then decreased appreciably with the increase of ultrasonic frequency (Fig. 1). This is due to the fact that phenomenon of cavities increases along with the enhancement of ultrasonic frequency, which accelerates the oil osmosis in Korean pine seed

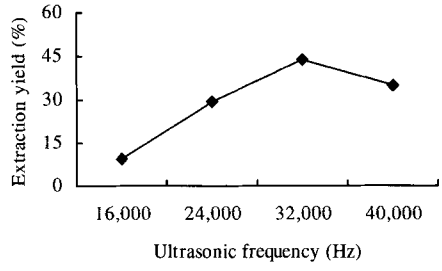

Fig. 1 Influence of ultrasonic frequency on extraction yield of seed oil of Korean pine

Effect of ultrasonic time on extraction yield

The effect of different ultrasonic time on extraction yield was detected at ultrasonic frequency of 24 000 Hz, the ratio of mate-

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rial to liquid of 1:10, and temperature of 40 ℃. The result showed that extraction yield increased with the increase of ultrasonic time, but after a certain time when osmotic-pressure of fat cell reached a balance, the extraction yield kept invariableness (Fig 2). The experiment result demonstrates that seed oil of Korean pine can be extracted fully within 40 min under ultrasonic treatment.

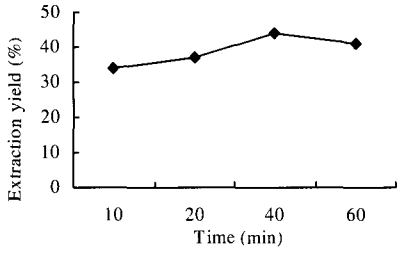

Fig. 2 Influence of ultrasonic time on extraction yield of seed oil of Korean pine

Effect of the ratio of material to liquid on extraction yield

The effect of different ratio of material to liquid on extraction yield was detected at ultrasonic frequency of 24 000 Hz, ultrasonic time of 20 min, and temperature of 40 ℃. Extraction yield increased with the increase of the ratio of material to liquid (Fig. 3). The extraction yield at the ratio of material to liquid of 1:40 was 7.4% higher than that at the ratio of 1:30. Since big ratio of seed to absolute alcohol might increase the difficulty in reclaiming alcohol, the ratio of material to liquid at 1:40 is suggested, according to the experimental result.

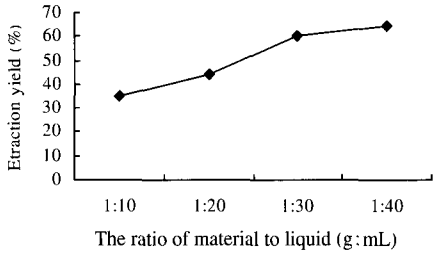

Fig.3 Influence of material liquid on extraction yield of seed oil of Korean pine

Effect of ultrasonic temperature on extraction yield

The effect of different ultrasonic temperature on extraction yield was investigated at ultrasonic frequency of 24 000 Hz, ultrasonic time of 20 min, and the ratio of material and liquid of 1:10. The extraction yield increased with the increase of temperature (Fig. 4). The yield of extraction at 80 ℃ increased by 3.3% compared with that at 70 ℃. The result showed that it is not necessary to keep on higher temperature during the extraction. Too high temperature will make a disadvantage for the stability of unsaturated fatty acid in Korean pine seed oil, thus the extraction temperature should keep under 80 ℃.

Synthetic factors

On the basis of experiment and analysis of single factor, the ultrasonic frequency, extracting temperature, ultrasonic time, and the ratio of material to liquid were selected separately to set up three levels experiments (Table 1), and the influence of each factor on extraction yield was observed. Design and results of orthogonal experiment were shown in Table 2.

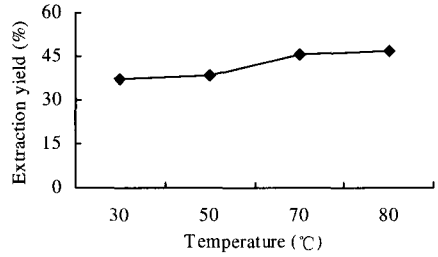

Fig.4 Influence of ultrasonic temperature on extraction yield of seed oil of Korean pine

Table 1. Factors and levels of orthogonal experiment

Levels	Factors			
	Temperature (℃)	Time (min)	Ration of material to liquid (g: mL)	Frequency (Hz)
1	60	30	1:20	28 000
2	70	40	1:30	32 000
3	80	50	1:40	36 000

Table 2. Design and results of orthogonal experiment L9(3^4)

No. of Test	A	B	C	D	Yield (%)
1	1	1	1	1	46.0
2	1	2	2	2	52.0
3	1	3	3	3	48.0
4	2	1	2	3	55.0
5	2	2	3	1	44.0
6	2	3	1	2	64.0
7	3	1	3	2	48.0
8	3	2	1	3	57.0
9	3	3	2	1	70.0
k1	48.667	49.667	55.667	53.333	
k2	54.333	51.000	59.000	54.667	
k3	58.333	60.667	46.667	53.333	
R	9.666	11.000	12.333	1.334	

Note: A—Temperature; B-- ultrasonic time; C--the ratio of material to liquid; D-- ultrasonic frequency.

From Table 2, we can draw a conclusion that the order of influence of different parameters on extraction yield is: ratio of material to liquid>Time>Temperature>Frequency. The optimum condition is $A_3B_3C_2D_2$, means that extracting temperature is 80℃, extracting time is 50 min, the ratio of material to liquid is 1:30, and frequency is 32 000 Hz. Variance analysis showed that Extracting temperature, time, and ratio of material to liquid also had remarkable influences on the extraction yield of Korean pine seed oil (Table 3).

Table 3. Variance analysis

Source of variance	SS	F	F_α	Significance
A	141.556	39.808	19.000	*
B	216.222	60.737	19.000	*
C	244.222	68.679	19.000	*
D	3.556	1.000	19.000	
Error	3.56			

Notes: A--Temperature; B-- ultrasonic time; C--the ratio of material to liquid; D-- ultrasonic frequency. All of freedom degree is 2, α=0.05. * represents significant difference.

Discussions

Ultrasonic technology has been applied widely in the extraction of effective components of many materials. Some experiments have been carried out to develop ultrasonic extraction method. Ultrasonic extraction has been regarded as a fast, reliable and inexpensive technique, and it is more applicable in the extraction of materials than the conventional techniques of extraction.

Our study demonstrates that ultrasound is a reliable and great efficiency tool for the fast extraction of Korean pine seed oil, and the optimized ultrasonic extraction parameters are extracting temperature of 80℃, ultrasonic time of 50 min, the ratio of material to liquid of l: 30, and ultrasonic frequency of 32 000 Hz. The most extraction yield is about 70%. .

Alcohol is chosen in the experiment because it is nontoxic, inexpensive, and safe during the production, but its boiling point is higher than that of common organic solvents. Therefore, comparison of alcohol with other organic solvent (ether, acetone, methyl alcohol, etc.) needs to be carried out later.

As temperature is the main factor in accelerating oxidization of oil, more attention should be paid to keeping a certain temperature condition in extraction process, such as the use of vacuum evaporation technology or antioxidant (Li *et al.* 2003). In this experiment we mainly concerned the yield of extraction, the quality of the extraction with different extracting temperature and other correlative problems need to go deep into research.

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